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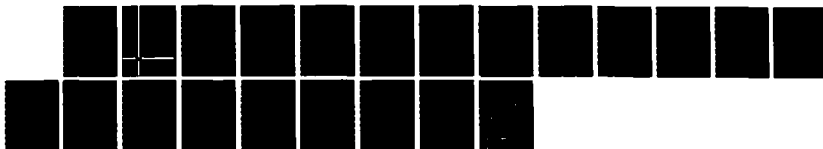
LABORATORY EVALUATION OF COMMERCIAL ANTIFREEZES(U) ARMY
BELVOIR RESEARCH DEVELOPMENT AND ENGINEERING CENTER
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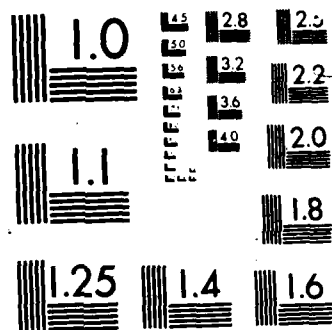
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Report 2429

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LABORATORY EVALUATION OF COMMERCIAL ANTIFREEZES

By
James H. Conley
Robert G. Jamison

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United States Army
Belvoir Research, Development & Engineering Center
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19. ABSTRACT (Continue on reverse if necessary and identify by block number) The object of this study was to investigate the potential corrosion problems when commercial antifreezes are mixed with each other and with the military antifreeze MIL-A-46153. Results show that corrosion-inhibiting properties of commercial antifreezes have been improved. Mixtures of commercial antifreezes, however, are still less than adequate.			
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LABORATORY EVALUATION OF COMMERCIAL ANTIFREEZES

I. INTRODUCTION

There has been a continuing trend for the Army to purchase commercial vehicles/equipment. These vehicles/equipment are covered by the manufacturers' warranties which require the use of specified, expendable materials. One such material is antifreeze.

Each vehicle manufacturer recommends the use of a particular product which is usually covered by the manufacturer's own specification. Equipment failure resulting from the use of a product not recommended by the manufacturer will negate the warranty. In every case, the judgment is made by the vehicle manufacturer.

Most current commercial antifreezes are compatible with each other from the standpoint of solubility (i.e., there is no precipitation of inhibitors when two different antifreezes are mixed). Occasionally, there are materials that visibly interact and form precipitates, but this is only one aspect of antifreeze compatibility. Mixtures which show no precipitation may still react with one another and form soluble compounds that are corrosive to the cooling system metals.

True compatibility only can be verified by a corrosion test such as the American Society for Testing and Materials (ASTM) Method D 1384, Corrosion Test for Engine Coolants in Glassware. Obviously, the number of possible combinations of two or more antifreeze fluids is infinite, and actual tests have to be limited to a reasonable number of mixtures.

In a previous study,¹ the corrosion test results indicated serious corrosion problems with mixtures. Because of the continuing thrust within DOD to use commercial products, this follow-on study was conducted. As stated earlier, the potential for admixture is very high, and the impact on vehicle warranties is significant. Further, there has been a push within NATO on development of a Guided Specification for *performance-type* antifreeze, and the type of data generated in this study is very important to support or challenge this effort. Most commercial antifreezes are based upon performance testing, whereas MIL-A-46153 is based strictly on a composition of proven satisfactory performance.

Eleven commonly-used antifreeze compounds (five factory-fill and six aftermarket) were selected to determine compatibility with each other and with the Army's MIL-A-46153² antifreeze in terms of corrosiveness, changes in reserve alkalinity (RA), and changes in acidity (pH factor), because these are the more important property aspects of antifreeze mixtures.

II. DETAILS OF TEST

All 11 commercial antifreezes and the MIL-A-46153 were analyzed by gas chromatography to identify the freeze point depressant and the approximate concentration. Identification of the major components of the inhibitor systems was accomplished by atomic absorption spectroscopy. No attempt was made to identify minor components.

¹ Conley, James H. and Jamison, Robert G., "Evaluation of Commercial Antifreezes," MERADCOM Report 2248, May 1978.

² Military Specification MIL-A-46153, Antifreeze, Ethylene Glycol, Inhibited, Heavy Duty, Single Package.

All corrosion tests were conducted according to ASTM Method D 1384, Corrosion Test for Engine Coolants in Glassware. This method describes a simple beaker test for evaluating the corrosive effects of engine coolants on metal specimens. Metal specimens typical of those present in automotive cooling systems are totally immersed in the test antifreeze solution using ASTM corrosive water with aeration for 336 hours at 88 °C (190 °F). The corrosion inhibitive properties of the test solution are evaluated on the basis of the weight changes incurred by the specimens. Each test was run in duplicate, and the average weight change was determined for each metal.

Tests on mixed antifreezes used two components in equal parts by volume diluted to 33 1/3 percent by volume with ASTM corrosive water containing 100 p/m each of chloride, sulfate, and bicarbonate.

Values for reserve alkalinity (RA) and acidity (pH) were measured on the packaged products and, also, measured on all other solutions before and after the corrosion tests. Reserve alkalinity of new antifreeze is used in production quality control and in specifications to indicate the amount of alkaline (basic) inhibitors present in the product. Similarly, the RA of used solutions is a measurement that indicates the amount of remaining alkaline inhibitors in coolant performance testing. The pH of a solution is commonly considered to be the negative logarithm (to the base 10) of the hydrogen ion concentration and, alone, is not a dependable indication of either effectiveness or remaining life of a solution. Both RA and pH measurements are effective in determining the presence of a buffer. A buffer is any substance or combination of substances which, when dissolved in water, produces a solution that resists a change in its hydrogen ion concentration upon the addition of acid or base. A considerable number of antifreezes, including MIL-A-46153, rely upon a buffer-type inhibitor for corrosion protection.

III. DISCUSSION OF RESULTS

Table 1 shows the comparative analysis of the 11 commercial antifreezes and MIL-A-46153. The gas chromatographic analysis shows that all products used ethylene glycol as the major freeze point depressant. Analysis by atomic absorption spectroscopy shows that all products, except Antifreeze G, contain boron. All 11 commercial products contain silicon. Antifreezes A, B, D, F, G, and MIL-A-46153 contain phosphorous. Antifreeze A contains a significant amount of molybdenum, and Antifreezes D and E contain 10 p/m of molybdenum. The pH values range from 6.36 to 11.01, and the RA values range from 6.0 to 18.2. The pH and RA values for MIL-A-46153 are 6.35 and 26.6, respectively.

The corrosion test results of the commercial products and the MIL-A-46153 are shown in Table 2. None of the products exceeded the recommended weight loss limit on any of the six metals. Four of the commercial products showed attack of one metal, and one showed attack of three metals. The MIL-A-46153 showed slight etching of the copper specimen. None of the 11 commercial antifreezes tested *good* with the Army's reserve alkalinity test strip. Reserve alkalinity values from 8 to 10 indicate *good*, 6 to 8 indicate *borderline*, and 4 to 6 indicate *poor; change coolant*. Four of the 11 commercial products showed *borderline* and seven showed *poor; change coolant*. The use of the test kit is detailed in TB-750-651³ and is based on the MIL-A-46153 formulation.

Table 3 shows the corrosion test results of 1:1 mixtures of MIL-A-46153 (Antifreeze M) with each of the 11 commercial products. All metal specimens passed the ASTM weight loss limit, but the aluminum specimen in Antifreezes M, D and M, K showed high weight losses and corrosion. Antifreeze M, L showed heavy etching of the

³ Technical Bulletin TB-750-651, Use of Antifreeze Solutions and Cleaning Compounds in Engine Cooling Systems.

Table 1. ANALYSIS OF PACKAGED PRODUCTS

Test No.		Gas Chromatography						Atomic Absorption Spectroscopy				
		Ethylene	Propylene	Cellosolve	Butyl Carbitol	Methyl Carbitol	As Packaged		ppm Boron	ppm Silicon	ppm Phosphorous	ppm Molybdenum
							pH	RA				
1	A	89.6	—	—	—	10.2	10.52	14.4	500	700	200	200
2	B	91.7	—	—	8.1	—	10.4	16.0	600	600	100	0
3	C	83.9	2.9	—	—	12.5	7.02	11.8	1400	100	0	0
4	D	95.1	3.5	—	—	—	6.36	6.0	2500	300	75	10
5	E	92.4	—	—	5.6	—	9.88	12.6	600	500	0	10
6	F	85.8	—	—	—	14.0	10.56	12.8	300	600	50	0
7	G	96.1	2.5	—	—	—	11.01	17.2	0	400	100	0
8	H	90.8	—	6.5	—	2.1	11.01	12.00	600	300	0	0
9	I	94.0	—	—	4.1	—	10.6	18.2	1800	500	0	0
10	K	93.0	—	4.0	—	—	10.77	12.8	1100	100	0	0
11	L	95.0	—	—	—	4.8	7.09	10.37	800	500	0	0
12	M	89.0	6.5	—	—	—	6.35	26.6	4700	0	100	0

Table 2. ASTM D 1384 CORROSION TEST RESULTS - PACKAGED PRODUCTS

Weight Loss Per Coupon in Milligrams

Test No.	Antifreeze	Copper	Solder	Brass	Steel	Cast Iron	Cast Aluminum	Before Test		After Test	
								pH	RA	pH	RA
1	A	4.28	8.86	5.45	1.01	2.33 ^b	0.98	10.59	4.6	10.15	4.8
2	B	1.97	11.53	4.00	1.21	1.34	0.68	10.69	5.6	10.07	5.6
3	C	3.83	6.86	4.41	0.58	0.02	1.94	8.34	4.8	7.87	3.2
4	D	3.25	6.93	3.65	1.17	0.73	5.42 ^c	7.98	6.4	7.95	5.2
5	E	2.41	7.74	4.37	0.63	1.10 ^d	1.19	10.38	4.8	9.42	4.2
6	F	3.83	13.25	4.87	0.62	1.76	0.93	10.71	5.0	9.93	4.4
7	G	3.69	5.00	5.05	0.73	2.44	2.81	10.94	6.0	10.27	5.4
8	H	3.10	7.90	4.60	0.25	2.30 ^e	2.79	10.61	4.0	9.86	4.6
9	I	3.15	11.45	5.40	0.50	0.27	1.39	10.21	6.4	9.39	6.4
10	K	3.43	0.51	3.34	0.27	0.27	0.02	10.68	4.4	9.73	4.4
11	L	3.72	8.79	4.43	0.79 ^d	1.36 ^d	7.09 ^e	10.20	7.2	9.35	8.0
12	M	3.86 ^a	18.86	7.58	0.74	2.36	0.85	7.95	8.2	7.78	10.2
ASTM Weight Loss Limit (maximum)		10	30	10	10	10	30				

NOTE: Appearance of Test Coupon:

- a - Slight Pitting
- b - Moderate Pitting
- c - Heavy Pitting

d - Electrolytic Stain
e - Heavy Pitting

f - Moderate Stain
g - Moderate Corrosion

**Table 3. ASTM D 1384 CORROSION TEST RESULTS -
50/50 MIXTURES OF MIL-A-46153/COMMERCIAL PRODUCT**

Test No.	Antifreeze Mixture	Weight Loss mg/Specimen									
		Copper	Solder	Brass	Steel	Cast Iron	Cast Aluminum	Before Test		After Test	
								pH	RA	pH	RA
1	NI A	5.97	8.86	9.34	1.30	3.15	1.62	8.09	6.4	8.12	7.2
2	NI B	4.74	8.85	5.33	1.20	1.10	5.37	8.13	6.8	8.19	7.2
3	NI C	4.55	11.11	4.75	2.32	1.13	2.82	7.96	6.0	7.90	7.0
4	NI D	3.18	10.46	4.59	0.77	0.48	27.35 ^c	7.96	7.2	7.91	8.2
5	NI E	2.42	7.67	5.94	1.57	1.28	1.44	8.12	7.2	8.16	8.0
6	NI F	5.48	11.00	4.99	1.01	2.01	1.13	8.09	6.4	8.10	7.2
7	NI G	2.93	9.30	5.72	1.24	1.08	1.47	8.13	7.6	8.00	9.6
8	NI H	4.27	7.06	7.60	1.18	1.37	3.10	8.00	6.4	8.05	7.6
9	NI I	3.14	9.72	5.70	1.32	2.08	3.87	8.03	6.4	8.02	6.8
10	NI K	7.56	8.35	8.54	0.93	1.43 ^d	21.53 ^c	8.02	6.4	7.97	7.4
11	NI L	4.30	10.27	5.62 ^c	0.98	1.04	4.77	8.12	7.4	8.19	8.2
ASTM Weight Loss Limit (maximum)		10	30	10	10	10	30				

* All an dispersion tubes had to be changed due to clogging.

NOTE: Appearance of Test Coupon:

- | | | |
|---------------------|----------------------|-----------------------|
| a. Slight Etching | d. Electrolytic Slam | f. Moderate Slam |
| b. Moderate Etching | e. Heavy Pitting | g. Moderate Corrosion |
| c. Heavy Etching | | |

**Table 4. ASTM D 1384 CORROSION TEST RESULTS -
50/50 MIXTURES OF ALL COMMERCIAL PRODUCTS***

(cont on pp 8-10)

Weight Loss mg/Specimen

Test No.	Antifreeze Mixture	Copper	Solder	Brass	Steel	Cast Iron	Cast Aluminum	Before Test		After Test	
								pH	RA	pH	RA
1	A B	17.82	14.38	8.13	2.28	4.97 ^a	5.12	10.69	6.6	10.23	4.0
2	A C	7.27	17.09	10.59	2.14	3.97	1.45	8.80	4.2	8.47	3.2
3	A D	6.92	11.43	8.07	1.82	6.02	2.08	8.45	5.0	8.60	4.6
4	A E	8.52	19.44	6.76	0.97	3.12 ^a	4.72	10.60	4.2	10.00	5.0
5	A F	5.26	12.20	5.75	1.16	1.59	0.07	10.51	4.0	10.00	4.2
6	A G	5.16	17.60	6.94	2.04	2.66	4.10	10.79	5.2	10.18	3.2
7	A H	6.17	11.75	8.07	1.81	4.63 ^a	8.06	10.72	4.4	10.02	4.0
8	A I	4.73	12.02	7.05	2.29	3.05	4.48	10.52	4.2	10.06	5.2
9	A K	6.45	11.66	7.20	1.75	3.46 ^a	3.78	10.71	4.4	10.15	4.0
10	A L	7.06	15.03	9.84	2.05	2.85	4.72	10.44	6.0	9.92	5.6
11	B C	4.12	15.95	5.63	1.97	2.77	1.73	8.91	4.2	8.58	4.0
12	B D	8.51	15.16	9.66	1.57	2.59	2.67	8.46	5.4	8.49	4.4
13	B E	5.81	16.25	5.79	1.35	3.61	3.27	10.31	4.6	9.84	4.2

* All air dispersion tubes had to be changed due to clogging.

NOTE: Appearance of Test Coupon

- a - Slight Etching
- b - Moderate Etching
- c - Heavy Etching

- d - Electrolytic Stain
- e - Heavy Pitting

- f - Moderate Stain
- g - Moderate Corrosion

brass specimen. All mixtures showed *borderline* with the Army's RA test strip. During the 14-day test, the air-dispersion tubes were replaced once a day or more because of coggling. The clogging was most likely caused by the silicate which undergoes a change during aeration, forming insoluble compounds.

Table 4 shows the corrosion test results of 1:1 mixtures of all commercial products. Test Numbers 1, 44, 50, and 54 failed the ASTM weight loss limit on one metal; and Test Numbers 52, 53, and 54 failed the ASTM weight loss limit on two metals. In addition, 15 of the 55 tests showed attack on one or more metal specimens. Test Numbers 1, 10, 34, 40, 47, 49, and 54 showed *borderline* with the Army's RA test strip. All of the remaining 48 tests showed *poor; change coolant*. Again, all air dispersion tubes had to be replaced at least once a day or more due to clogging.

IV. CONCLUSIONS

From the results listed in this study (as compared to a similar study conducted in 1978), it is evident that commercial antifreezes have been improved significantly. It is also noted that the use of silicate inhibitors has become more prevalent and has improved the corrosion protection of cooling system metals. Admixing of products that are not identical in composition will always present serious corrosion problems. Twenty-seven percent of the mixtures tested produced corrosion on one or more of the cooling system metals. It is also clear that the use of these mixtures will render the RA test kit unusable, leaving the troops in the field with no method of adequately maintaining vehicle cooling systems. Use of the MIL-A-53009⁴ is questionable since it was designed specifically for reinhibiting MIL-A-46153.

It is concluded, therefore, that the use of commercial antifreezes in military vehicles is not recommended. In instances where a vehicle warranty includes the use of a proprietary factory-fill antifreeze other than MIL-A-46153, only the manufacturer's recommended antifreeze should be used during the warranty period. Immediately after expiration of the warranty period, the cooling system should be drained, flushed, and refilled with MIL-A-46153 antifreeze, only.

⁴ Military Specification MIL-A-53009, Additive, Antifreeze Extender, Liquid Cooling System.

Table 4 (cont). ASTM D 1384 CORROSION TEST RESULTS -
50/50 MIXTURES OF ALL COMMERCIAL PRODUCTS*

Weight Loss mg/Specimen

Test No.	Antifreeze Mixture	Copper	Solder	Brass	Steel	Cast Iron	Cast Aluminum	Before Test		After Test	
								pH	RA	pH	RA
14	B I	3.06	8.77	5.85	1.81	0.58	1.96	10.54	4.4	10.01	5.0
15	B G	5.19	16.11	7.03	2.04	1.80	4.47	10.58	5.2	10.15	5.0
16	B H	3.72	9.60	7.50	1.44	3.00	1.81	10.40	4.6	10.03	4.6
17	B I	3.36	11.63	5.37	1.01	2.47	3.09	10.23	5.6	10.00	6.2
18	B K	4.80	11.10	6.16	1.93	1.53	3.42	10.44	4.8	9.96	4.8
19	B I	2.84	10.68	7.13	1.16	3.66	5.22	10.26	6.2	9.97	6.4
20	C D	9.65	4.49	8.67	0.86	7.27	2.12	8.24	4.4	8.03	4.06
21	C I	5.37	23.30	6.18	0.57	1.95	1.35	8.72	3.8	8.54	3.6
22	C I	3.27	7.59	3.30	0.82	0.12	0.71	8.79	3.8	8.24	4.2
23	C G	4.41	13.47	4.97	1.51	1.86	4.19	9.12	4.4	9.01	2.4
24	C H	4.31	9.47	4.39	1.08	1.39	Nil	8.81	3.6	8.55	3.8
25	C I	3.73	13.98	5.42	0.75	5.36	2.41	8.87	4.9	8.54	5.2
26	C K	4.06	10.58	4.09	0.82	1.27	2.76	8.80	3.9	8.52	4.0
27	C I	2.92	13.11	6.14	1.28	5.23	3.29	8.79	5.2	8.74	5.6
28	D I	3.81	12.31	4.92	1.78	3.62	2.70	8.35	5.0	8.36	6.0

*All anti dispersion tubes had to be changed due to clogging.

Table 4 (cont). ASTM D 1384 CORROSION TEST RESULTS -
50/50 MIXTURES OF ALL COMMERCIAL PRODUCTS*

Test No.	Antifreeze Mixture	Weight Loss mg/Specimen									
		Copper	Solder	Brass	Steel	Cast Iron	Cast Aluminum	Before Test		After Test	
								pH	RA	pH	RA
29	D/F	5.06	9.44	6.11	2.27	1.03	5.16 ^{b,f}	8.32	4.8	8.16	5.4
30	D/G	5.91	11.09	6.11	1.26	5.25	4.40	8.48	5.4	8.51	6.4
31	D/H	4.62	Nil	5.04	1.12	2.83	2.69	8.37	5.0	8.42	6.0
32	D/J	5.15	9.16	5.73	1.57	3.18	6.20	8.44	5.8	8.40	7.0
33	D/K	4.54	14.05	4.98	1.33	2.90	7.68 ^{f,g}	8.34	5.0	8.42	5.6
34	D/L	5.09	13.87	5.07	1.64	1.46	4.99	8.42	6.2	8.69	7.6
35	F/F	3.08	8.14	4.19	1.61	0.95 ^d	3.35 ^{e,f}	10.49	4.0	9.71	4.0
36	F/G	3.14	12.39	3.12	0.68	2.33	3.97	10.67	4.8	10.07	4.6
37	F/H	3.92	9.07	3.83	+ 3.39	1.87	0.48	10.45	4.6	9.85	4.4
38	F/J	4.22	11.53	5.87	1.38	0.80	2.25	10.12	5.4	9.70	5.8
39	F/K	5.63	15.73	5.37	1.31	1.09	4.60 ^{e,f}	10.39	4.2	9.62	4.4
40	F/I	3.02	9.74	5.48	1.79	0.79	4.09	10.23	6.0	9.76	6.6
41	F/G	3.55	8.25	4.27	1.16	0.98	5.72 ^e	10.68	4.4	10.07	4.6
ASTM Weight Loss Limit (maximum)		10	30	10	10	10	30				

*All air dispersion tubes had to be changed due to clogging.

NOTE: Appearance of Test Coupon:

a - Slight Pitting
b - Moderate Pitting
c - Heavy Pitting

d - Electrolytic Stain
e - Heavy Pitting

f - Moderate Stain
g - Moderate Corrosion

Table 4 (cont). ASTM D 1384 CORROSION TEST RESULTS -
50/50 MIXTURES OF ALL COMMERCIAL PRODUCTS*

Test No.	Antifreeze Mixture	Weight Loss mg/Specimen									
		Copper	Solder	Brass	Steel	Cast Iron	Cast Aluminum	Before Test		After Test	
								pH	RA	pH	RA
42	I-H	3.10	11.27	4.89	0.80	2.76 ^b	3.27 ^b	10.61	4.0	9.80	4.0
43	I-J	2.92	7.30	3.43	0.61	0.87	8.64 ^b	10.38	5.2	9.54	5.2
44	I-K	3.46	8.90	5.26	0.70	1.18 ^b	36.30 ^b	10.58	4.0	9.67	4.0
45	I-L	3.77	8.60	4.57	0.97	1.10	6.37 ^b	10.40	5.6	9.90	5.8
46	G-H	3.47	9.25	5.92	1.55	5.32	4.76	10.69	4.6	10.07	4.0
47	G-J	3.73	7.72	7.71	2.10	1.74	3.41	10.43	6.4	9.98	4.9
48	G-K	3.30	7.84	4.15	1.47	3.38	8.08	10.46	5.2	10.22	4.1
49	G-L	1.72	5.38	3.06	0.99	1.94	5.62	10.58	6.2	9.99	5.7
50	H-J	6.69	36.64	8.99	2.26	4.15 ^d	9.34	10.26	5.2	9.96	5.4
51	H-K	8.13	4.73	6.61	3.00	1.84	7.48	10.51	4.0	9.80	3.8
52	H-L	8.44	43.57	11.79	3.78	4.77	15.19	10.37	4.4	9.71	5.6
53	J-K	5.82	35.19	7.53	2.86	3.91	66.33 ^e	10.35	5.0	9.73	5.4
54	J-L	8.54	39.18	10.13	3.17	4.23	19.09	10.15	6.6	9.88	7.2
55	K-L	11.72	48.14	13.01	3.61	7.93	21.10	10.40	5.6	9.99	5.5
ASTM Weight Loss Limit (maximum)		10	30	10	10	10	30				

*All air dispersion tubes had to be changed due to clogging.

NOTE - Appearance of Test Coupon:

- a - Slight Etching
- b - Moderate Etching
- c - Heavy Etching

- d - Electrolytic Stain
- e - Heavy Pitting

- f - Moderate Stain
- g - Moderate Corrosion

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